Failure effects in Oxide Ceramic Matrix Composites with defects caused by the laminating process

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Abstract

Oxide Ceramic Matrix Composites - produced by the Pritzkow Spezialkeramik company – are based on ceramic endless fibres, such as 3M™ Nextel™ 610, and matrix materials with content of Al_2O_3, SiO_2, mullite and 8YSZ (8 wt-% yttrium stabilised zirconia). The processing can be described as impregnating the ceramic textile with the ceramic slurry by knife coating. In this processing, defects like trapped air, agglomerates of slurry, damages of the textile and chopped fibre strands can be found. Defects can also occur as result of the laminate design. These defects will be described in this paper.

Structures can have insufficient mechanical properties in the region of the defects. Therefore more information is needed about the effects caused by material defects. The results of these investigations will be very helpful in the design of mechanically loaded structures.

In this paper, produced effects on the material properties by the described defects caused by the laminating process and the laminate design are discussed. Mainly the influence on the mechanical properties will be shown by the 4-point bending test.

The different non-destructive testing methods such as

- vibrometry (analysis of frequency)
- ultrasound
- thermography
- computer tomography

are used to detect the internal defects, as delamination and internal material damages.

1. Introduction

For many industrial applications of Ceramic Matrix Composites (CMCs) long term high temperature stability in oxidizing atmosphere is required. In addition a good thermal shock resistance and a damage tolerant fracture behaviour is requested. Therefore the best combination for CMCs is an oxide fibre and an oxide ceramic matrix. With 3M™ Nextel™ 610 fabric and a matrix based on Al_2O_3-powder and a 8YSZ-binder, developed by the Fraunhofer-ISC [1] an Oxide Ceramic Matrix Composite (OCMC) with a porous and weak matrix was designed by the Pritzkow Spezialkeramik. The microstructural design is comparable to the design of Levi et al. [2] shown in Fig. 1.

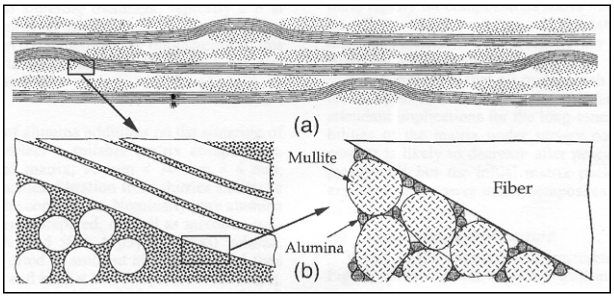

Fig. 1. Microstructural design of a OCMC with a matrix based on mullite-powder and an Al_2O_3-binder [2]

The samples for testing the mechanical properties of structures with defects were produced with 3M™ Nextel™ 610 fibres woven as eight-harness satin fabric which are desized. The fabric is impregnated with an Al_2O_3–8YSZ slurry by knife coating. The layers are stacked and pressed together to 4-layer plates. The sintering process is followed by an infiltration with an 8YSZ-sol and a second sintering process. A cross section of a sample without defect is shown in Fig. 2.

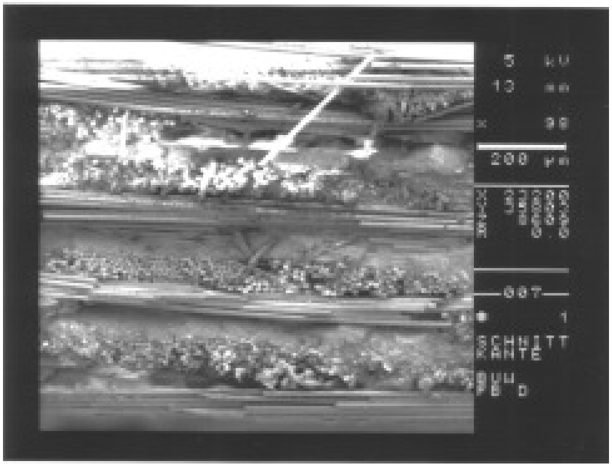

Fig. 2. SEM-micrograph of an unpolished 4-layer OCMC sample

OCMCs based on an Al_2O_3–8YSZ matrix are used in various industrial processes. Examples are:

- lift gate for a furnace with a H_2/N_2-atmosphere to produce sintered metal components (Fig. 3)
- sintering aid
- tubes for casting
- furnace components like splinter shield
- burner components like flame tubes
- gas turbine components.

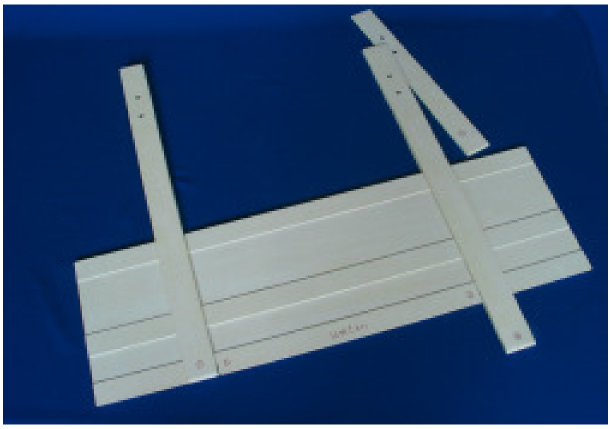

Fig. 3. Lift gate for a furnace with a H_2/N_2-atmosphere

2. Defects

In simple structures mainly two different types of defects (Fig. 4) are seen.

1. The area of the beginning and the end of a fabric based wound tube,
2. areas where the fabric of the internal layers have to be cut.

Fig. 4. Defect type 1 (left) and type 2 (right) on a tube with flange

These defects will be the main theme of this investigation.

2.1 Defect Type 1 (wound tube with overlap)

The defect type 1 will occur when a tube is fabricated based on fabric wound on a cylindrical mould. The wound tube has a beginning and an end of the fabric like shown in the schema (Fig. 5). Both ends can be in one line which means a length of the overlap l_F of zero. If the length of the overlap l_F is positive a short part will have one additional layer. In the other case the length of the overlap l_F is negative, which means that for a short distance one layer is missing.

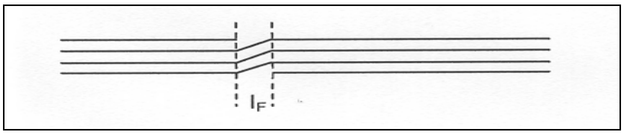

Fig. 5. Defect type 1, wound tube with overlap

2.2 Defect Type 2 (internal cut)

The defect type 2 will occur for example when the flange of a tube is fabricated (Fig. 4) because there is not enough fabric to produce the ring. This part will be filled in this case with a triangle of fabric. The internal defect can be described by the distance l_F of the internal cut of layer 2 and 3 in a 4-layer plate. The defect length l_F can be 0 up to infinite.

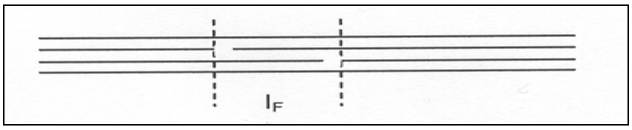

Fig. 6. Defect type 2, internal cut

3. Experimental

Samples with defects of type 1 (wound tube with overlap) and type 2 (internal cut) were produced with different defect lengths l_F. For each type of defect a reference sample without any defect is produced. These samples (4-layer laminate with a thickness of 0,95mm) have been tested by 4-point-bending [3]. The load elongation curve of a reference sample (Fig. 7) shows the theory [4]. An OCMC is breaking in the first part linear elastic, then first cracks in the matrix will occur. The third part is dominated by the fibre reaching its maximum bending strength. At the end of the curve the mechanisms fibre pullout and debonding become visible (Fig. 8).

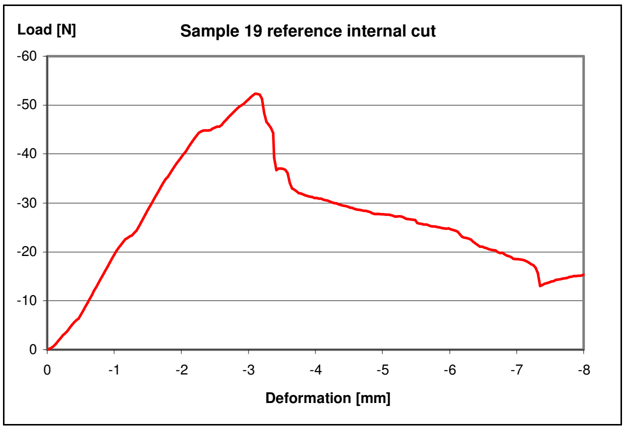

Fig. 7. Load elongation curve of a reference sample

Fig. 8. Fracture surface of a reference sample

3.1 Results of defect Type 1 (wound tube with overlap)

Samples of defect type wound tube with overlap having defect lengths l_F of +10 mm, +5 mm, 0 mm, -5 mm, -10 mm were tested. In the 4-point-bending test the reference thickness for calculating the bending strength is the thickness of the plate beside the defect. The result of the 4-point bending test (Fig. 9) shows several effects.

- The bending strength of all samples with a defect is lower than the bending strength of the reference sample.
- The smallest reduction of the bending strength is within a defect length of 0 mm to + 5 mm.
- The negative defect length, where one layer is missing, shows not a large negative effect on the bending strength as expected.
- The failure can be described as a delamination starting at the open end of the fabric. This is an effect due to the weak matrix.

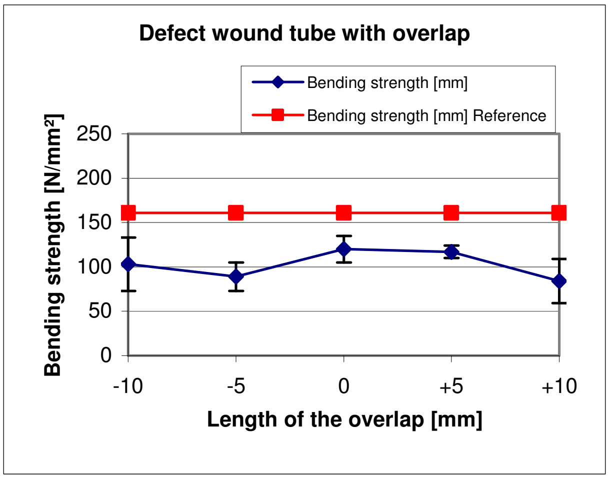

Fig. 9. Bending strength of samples with the defect wound tube with overlap

3.2 Results of defect Type 2 (internal cut)

Samples of the defect type internal cut having defect lengths l_F between 0 mm and 15 mm were tested. In the 4-point-bending test the reference thickness for calculating the bending strength is the thickness of the plate beside the defect. The result of the 4-point bending test (Fig. 10) shows no distinctive effect. The values of the bending strength of the reference sample are a bit lower than these of the samples with the internal cut. This means that an internal cut has no negative influence for a structure.

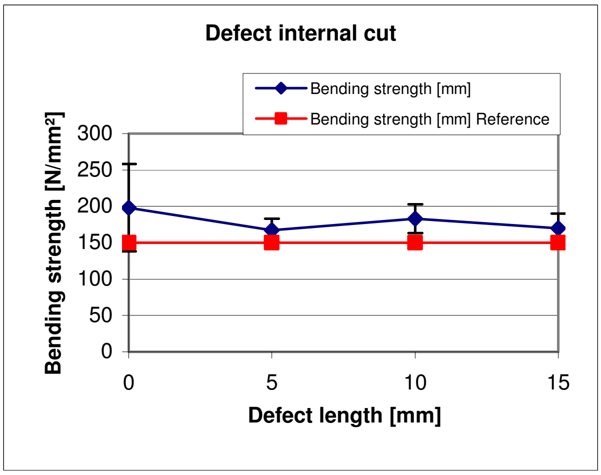

Fig. 10. Bending strength of samples with the defect internal cut

4. Non-Destructive Testing

For finding defects in structures based on OCMCs different non-destructive testing methods [5] can be used. These methods are:

- vibrometry (analysis of frequency)
- ultrasound
- thermography
- computer tomography.

4.1 Vibrometry

The oldest test method known and used in the porcelain industry is the acoustic emission test. To see if this method can be used for OCMCs primary tests have been done [6]. The reference sample shows two main frequencies (Fig. 11) characterising the fibre and the matrix. The other detections are probably very small defects which can occur in an OCMC.

The sample with the internal cut shows the same main frequencies and also two other frequencies (Fig.12). The higher frequencies detect the defects in the material, which indicates at the cuts in layer 2 and 3 of the 4-layer plate. This test is good to hear that there are defects but does not show their location.

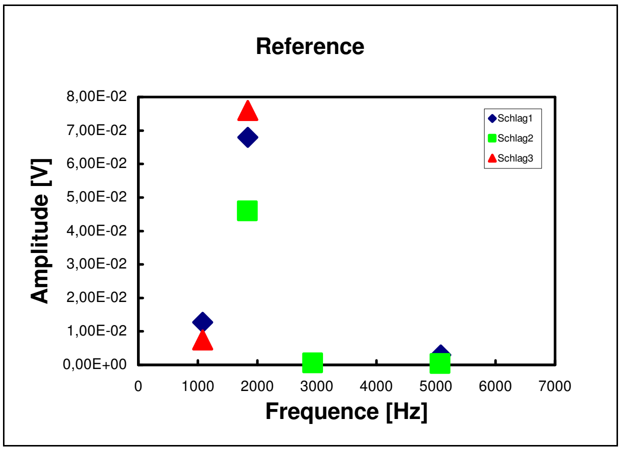

Fig. 11. Acoustic emission test, reference sample [6]

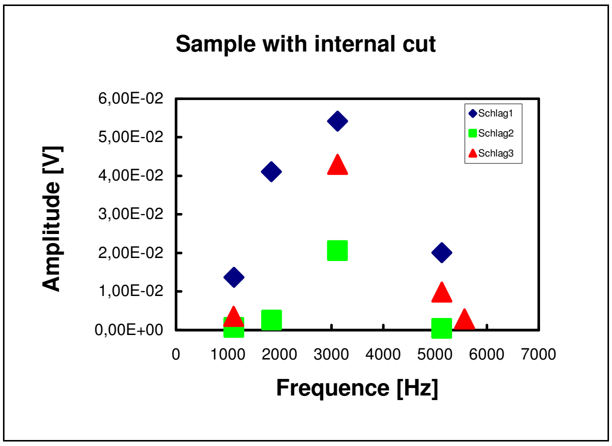

Fig. 12. Acoustic emission test of a sample with internal cut [6]

4.2 Ultrasound

To detect delaminations in a CMC or an OCMC ultrasonic test were mainly used. Fig. 13 shows that there are no delaminations in the reference sample. A sample with an internal cut has been tested under the same conditions. These cuts are parallel to the long side. In the ultrasonic test no delaminations were found, seen in Fig. 14. But also the defects can not be detected.

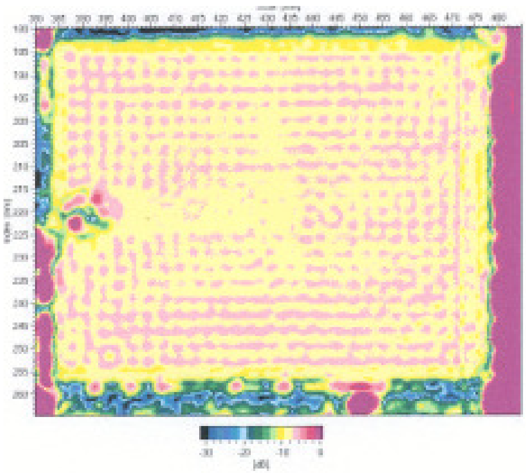

Fig. 13. Ultrasonic test on a reference sample [7]

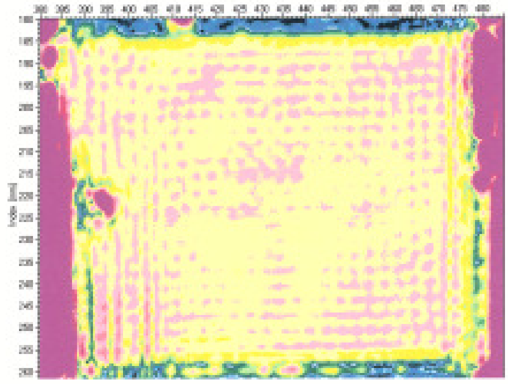

Fig. 14. Ultrasonic test on a sample with internal cut [7]

4.3 Thermography

The thermography is another testing method to detect defects in an OCMC. With this method delaminations can be found. The air locked in the delamination will reduce the heat conductance in the area of this defect. This area detect another temperature than the surrounding. Inhomogeneities in the wall thickness like produced in the defect type 1 (wound tube with overlap) also can be detected. That means for example a 4-layer sample with a negative overlap will have only 3-layers in the defect area. In this described area the heat transition is better, this results in different emission compared to the surrounding. If there is a defect type 2 (internal cut) where the defect length is less then 0,2 mm, no temperature inhomogeneity is seen.

4.4 Computer tomography

A new method for testing ceramics is the computer tomography (CT). This method is good for showing details like the internal material design mainly the ceramic material and the pores of this OCMC with a weak and porous matrix (Fig. 15). The CT can show the defects (Fig. 16) if the resolution is very detailed.

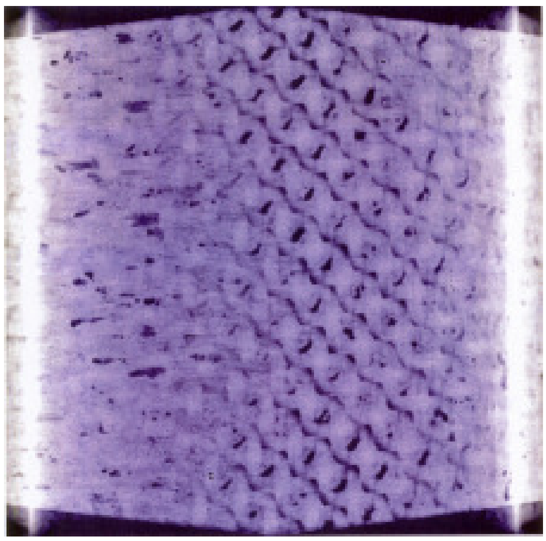

Fig. 15. CT of a reference sample, cut parallel to the surface [8]

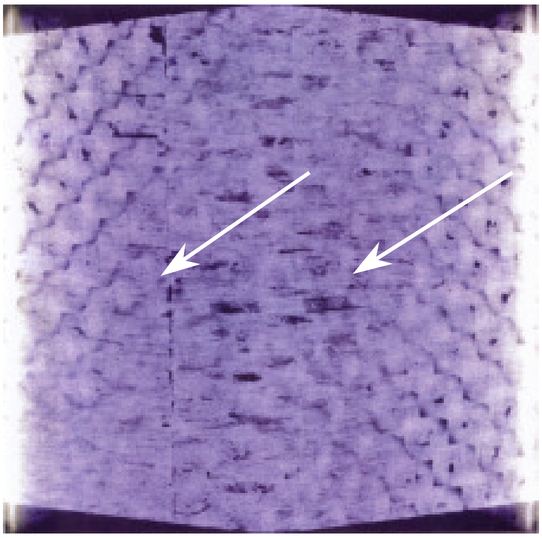

Fig. 16. CT of a sample with internal cut, the CT cut parallel to the surface. Both cuts of the fabric can be seen [8]

5. Conclusions

The types of defects produced in an OCMC structure are mainly described as wound tube (type 1) with overlap and internal cut (type 2).

The defect type wound tube with overlap (type 1) shows in the 4-point bending test that the beginning and the end of the wounded textile is a weak spot. There were the same effects M.G. Holmquist et al. [9] had been confronted testing pressurized tubes. The tested tubes also delaminated at the end of the wounded laminate.

The defect internal cut (type 2) shows that a destroyed fibre or fabric do not reduce the strength of an OCMC. This is comparable with the design study of R. Schäuble et al. [10], in which he states, that a good damage tolerance can be achieved by weak spots in the fibre and the matrix. It also shows, that an inhomogeneous matrix is positive for this type of material. An internal chaos is positive for the bending strength of a Ceramic Matrix Composite with a weak matrix.

The defect internal cut (type 2), which obviously do not have a negative influence on the mechanical properties can not be detected by ultrasonic test nor than by thermography.

6. References

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